

FORM PCT 1390

U.S. DEPARTMENT OF COMMERCE PATENT AND TRADEMARK OFFICE

REV. 5/93

TRANSMITTAL LETTER TO THE UNITED STATES
DESIGNATED/ELECTED OFFICE (DO/EO/US)
CONCERNING A FILING UNDER 35 U.S.C. 371

ATTORNEY'S DOCKET NO.

MATANO ET AL-3 PCT

U.S. APPLICATION NO. (if known, see 37 CFR 1.5)

09/914116

INTERNATIONAL APPLICATION NO.
PCT/JP00/00714

INTERNATIONAL FILING DATE
9 FEBRUARY 2000

PRIORITY DATE CLAIMED
24 FEB. 1999, 1 MAR. 1999,
1 MAR. 1999, 24 NOV. 1999

TITLE OF INVENTION

TEMPERATURE COMPENSATION MEMBER AND OPTICAL COMMUNICATION DEVICE SUING THE SAME

APPLICANT(S) FOR DO/EO/US

TAKAHIRO MATANO ET AL

Applicant herewith submits to the United States Designated/Elected Office (DO/EO/US) the following items and other information:

1. ☒ This is a **FIRST** submission of items concerning a filing under 35 U.S.C. 371.
2. ☐ This is a **SECOND** or **SUBSEQUENT** submission of items concerning a filing under 35 U.S.C. 371.
3. ☒ This is an express request to begin national examination procedures (35 U.S.C. 371 (f)) at any time rather than delay examination until the expiration of the applicable time limit set in 35 U.S.C. 371(b) and PCT Articles 22 and 39(I).
4. ☒ A proper Demand for International Preliminary Examination was made by the 19th month from the earliest claimed priority date.
5. ☒ A copy of the International Application as filed (35 U.S.C. 371(c)(2))
 - a. ☒ is transmitted herewith (required only if not transmitted by the International Bureau)
 - b. ☐ has been transmitted by the International Bureau.
 - c. ☐ is not required, as the application was filed in the United States Receiving Office (RO/US).
6. ☒ A translation of the International Application into English (35 U.S.C. 371(c)(2)).
7. ☐ Amendments to the claims of the International Application under PCT Article 19 (35 U.S.C. 371(c)(3)).
 - a. ☐ are transmitted herewith (required only if not transmitted by the International Bureau).
 - b. ☐ have been transmitted by the International Bureau.
 - c. ☐ have not been made; however, the time limit for making such amendments has **NOT** expired.
 - d. ☐ have not been made and will not be made.
8. ☐ A translation of the amendments to the claims under PCT Article 19 (35 U.S.C. 371(c)(3)).
9. ☒ An oath or declaration of the inventor(s) (35 U.S.C. 371(c)(4)).
10. ☐ A translation of the annexes to the International Preliminary Examination Report under PCT Article 36 (35 U.S.C. 371(c)(5)).

Items 11. to 16. below concern other document(s) or information included:

11. ☒ An Information Disclosure Statement under 37 CFR 1.97 and 1.98.
12. ☒ An assignment document for recording. A separate cover sheet in compliance with 37 CFR 3.28 and 3.31 is included.
13. ☒ A **FIRST** preliminary amendment.
☐ A **SECOND** or **SUBSEQUENT** preliminary amendment.
14. ☐ A substitute specification.
15. ☐ A change of power of attorney and/or address letter.
16. ☒ Other items or information:

PCT/ISA/210 - Int'l. Search Report (English)

1 sheet of formal drawings

Applicant Claims Priority under 35 U.S.C. §119 of JAPAN Application Nos. JP 47022/1999, 52780/1999, 52793/1999, 332577/1999,
filed on: FEBRUARY 24, 1999, MARCH 1, 1999, MARCH 1, 1999 AND NOVEMBER 24, 1999, RESPECTIVELY.
Applicant Claims Priority under 35 U.S.C. §120 of: PCT No. PCT/JP00/00714, filed on: FEBRUARY 9, 2000.

APPLICATION NO. (if known, see 37 CFR 1.5)

09/914116

INTERNATIONAL APPLICATION NO
PCT/JP00/00714ATTORNEY'S DOCKET NO
MATANO ET AL-3 PCT☒ The following fees are submitted:

Basic National Fee (37 CFR 1.492(a)(1)-(5)):

Search Report has been prepared by the EPO or JPO.....\$860.00

International preliminary examination fee paid to USPTO (37 CFR 1.482)
.....\$690.00Neither international preliminary examination fee paid (37 CFR 1.82) nor
international search fee (37 CFR 1.445(a)(2)) paid to USPTO.....\$1,000.00International preliminary examination fee paid to USPTO (37 CFR 1.482)
and all claims satisfied provisions of PCT Article 33(2)-(4).....\$100

ENTER APPROPRIATE BASIC FEE AMOUNT =

Surcharge of \$130.00 for furnishing the oath or declaration later than ____ 20 ____ 30
months from the earliest claimed priority date (37 CFR 1.492(e)).

CALCULATIONS

PTO USE ONLY

\$ 860.00

Claims

Number Filed

Number Extra

Rate

Total Claims

10 - 20 =

- 0 -

X \$18.00

\$

Independent Claims

4 - 3 =

- 1 -

X \$80.00

\$

80.00

Multiple dependent claim(s) (if applicable)

+ \$270.00

\$

TOTAL OF ABOVE CALCULATIONS =

\$ 940.00

Reduction by 1/2 for Small Entity status, if applicable.

\$

SUBTOTAL =

\$ 940.00

Processing fee of \$130.00 for furnishing the English translation later than ____ 20 ____ 30
months from the earliest claimed priority date (37 CFR 1.492(f)).

\$

TOTAL NATIONAL FEE =

\$ 940.00

Fee for recording the enclosed assignment (37 CFR 1.21(h)). The assignment must be
accompanied by an appropriate cover sheet (37 CFR 3.28, 3.31). \$40.00 per property +See cover sheet attached to assign
\$ to be charged to Deposit Acct

TOTAL FEES ENCLOSED =

\$ 940.00

Amount to be:
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\$

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\$

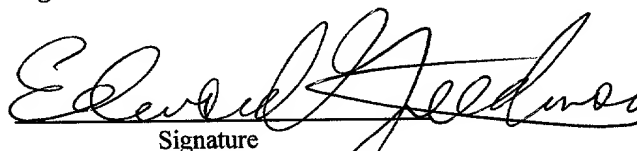
Applicant claims Small Entity status.

- a. ☒ A check in the amount of \$ 940.00 to cover the above fees is enclosed.
- b. ☐ Please charge my Deposit Account No. 03-2468 in the amount of \$ _____ to cover the above fees. A duplicate copy of this sheet is enclosed.
- c. ☒ The Commissioner is hereby authorized to charge any additional fees which may be required, or credit any overpayment, to Deposit Account No. 03-2468. A duplicate copy of this sheet is enclosed.

NOTE: Where an appropriate time limit under 37 CFR 1.494 or 1.495 has not been met, a petition to revive (37 CFR 1.137(a) or (b)) must be filed and granted to restore the application to pending status.

SEND ALL CORRESPONDENCE TO:

COLLARD & ROE, P.C.
1077 Northern Boulevard
Roslyn, New York 11576-1696
(516) 365-9802



Signature

Edward R. Freedman
Reg. No. 26,048

Express Mail No. EL 871 447 266 US

Date of Deposit August 23, 2001

I hereby certify that this paper or fee is being deposited with the United States Postal Service "Express Mail Post Office to Addressee" service under 37 CFR 1.10, on the date indicated above, and is addressed to the Ass't. Commissioner for Patents, Washington, D.C. 20231


Ingrid Mittendorf

PATENT

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

APPLICANT: TAKAHIRO MATANO ET AL
PCT NO.: PCT/JP00/00714 PCT FILED: 9 FEBRUARY 2000
PRIORITY: JP 47022/1999 PRIORITY FILED: 24 FEBRUARY 1999
and follows.
TITLE: Temperature Compensation Member and Optical Communication
Device Using the Same

PRELIMINARY AMENDMENT

ATTN.: BOX PCT APPLICATION

Ass't. Commissioner for Patents
Washington, D.C. 20231

Dear Sir:

Preliminary to the initial Office Action, please amend the
above-identified application as follows:

IN THE SPECIFICATION:

On Page 1, line 1, please insert the following paragraphs:

--CROSS REFERENCE TO RELATED APPLICATIONS

Applicants claim priority under 35 U.S.C. §119 of Japanese
Application Nos. JP 47022/1999, JP 52780/1999, JP 52793/1999,
JP 332577/1999, filed on February 24, 1999, March 1, 1999, March 1,
1999 and November 24, 1999, respectively. Applicants also claim
priority under 35 U.S.C. §120 of PCT/JP00/00714, filed on February
9, 2000. The international application under PCT article 21(2) was
not published in English.--

REMARKS

By this Preliminary Amendment, the application has been amended to conform with U.S. practice, the cross-reference to the related application has been inserted on page 1.

No new matter has been introduced.

Entry of this amendment is respectfully requested.

Respectfully submitted,

TAKAHIRO MATANO ET AL

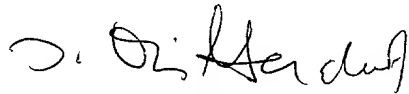


Allison C. Collard, Reg. No. 22,532
Edward R. Freedman, Reg. No. 26,048
Attorneys for Applicants

COLLARD & ROE, P.C.
1077 Northern Boulevard
Roslyn, New York 11576
(516) 365-9802

Express Mail No. EL 871 447 266 US
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Ingrid Mittendorf

1/pts

SPECIFICATION

TEMPERATURE COMPENSATION MEMBER AND OPTICAL COMMUNICATION DEVICE USING THE SAME

Technical Field

This invention relates to a temperature compensation member having a negative coefficient of thermal expansion and an optical communication device using the same.

Background Art

With the advance of the optical communication technology, a network using optical fibers has been rapidly built up. In the network, a wavelength multiplexing technique of collectively transmitting light beams having a plurality of different wavelengths has come into use, and a wavelength filter, a coupler, a waveguide, and the like have become important devices.

Some of the devices of the type described are changed in characteristics depending upon the temperature and may therefore cause troubles if used in the outdoors. This requires a technique for keeping the characteristics of these devices fixed or unchanged regardless of a temperature change, i.e., a so-called temperature compensation technique.

As a typical optical communication device which requires temperature compensation, there is a fiber Bragg grating (hereinbelow referred to as FBG). The FBG is a device in which a portion varied in refractive index in a grating-like pattern, i.e., a so-called grating is formed within a core of an optical fiber, and has a characteristic of reflecting a light beam having a specific wavelength according to the relationship represented by the following formula (1). Therefore, the device attracts attention as an important optical device in the

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optical communication system using a wavelength division multiplex transmission technique in which optical signals different in wavelength are multiplexed and transmitted through a single optical fiber.

$$\lambda = 2n\Lambda \quad \dots (1)$$

Herein, λ represents a reflection wavelength, n , an effective refractive index of the core, and Λ , a grating period of the portion varied in refractive index in the grating-like pattern.

However, the above-mentioned FBG has a problem that the reflection wavelength will be varied following the change in ambient temperature. The temperature dependence of the reflection wavelength is represented by the following formula (2) which is obtained by differentiating the formula 1 with the temperature T .

$$\begin{aligned} \partial \lambda / \partial T &= 2[(\partial n / \partial T) \Lambda + n(\partial \Lambda / \partial T)] \\ &= 2\Lambda[(\partial n / \partial T) + n(\partial \Lambda / \partial T) / \Lambda] \quad \dots (2) \end{aligned}$$

The second term of the right side of the formula (2), i.e., $(\partial \Lambda / \partial T) / \Lambda$ corresponds to a coefficient of thermal expansion of the optical fiber and has a value approximately equal to $0.6 \times 10^{-6}/^{\circ}\text{C}$. On the other hand, the first term of the right side corresponds to the temperature dependency of the refractive index of the core of the optical fiber and has a value approximately equal to $7.5 \times 10^{-6}/^{\circ}\text{C}$. Thus, it will be understood that the temperature dependency of the reflection wavelength depends upon both the variation in refractive index of the core and the change in grating period due to the thermal expansion but mostly results from the temperature-dependent variation of the refractive index.

As means for avoiding the above-mentioned variation in reflection wavelength, there is known a method in which the FBG is applied with tension depending upon the temperature change to thereby change the grating period so that a component resulting from the variation in refractive index is cancelled.

As a specific example of the above-mentioned method, proposal is made of a method in which the FBG is fixed to a temperature compensation member which comprises a combination of a material, such as an alloy or a silica glass, having a small coefficient of thermal expansion and a metal, such as aluminum, having a large coefficient of thermal expansion. Specifically, as illustrated in Fig. 1, an Invar (Registered Trademark) bar 10 having a small coefficient of thermal expansion has opposite ends provided with aluminum brackets 11a and 11b having a relatively large coefficient of thermal expansion attached thereto, respectively. An optical fiber 13 is fixed to the aluminum brackets 11a and 11b by the use of clasps 12a and 12b so that the optical fiber is stretched under a predetermined tension. At this time, adjustment is made so that a grating portion 13a of the optical fiber 13 is located between the two clasps 12a and 12b.

If the ambient temperature rises in the above-mentioned state, the aluminum brackets 11a and 11b are expanded to reduce the distance between the two clasps 12a and 12b so that the tension applied to the grating portion 13a of the optical fiber 13 is decreased. On the other hand, as the ambient temperature falls, the aluminum brackets 11a and 11b are contracted to increase the distance between the two clasps 12a and 12b so that the tension applied to the grating portion 13a of the optical fiber 13 is increased. Thus, by changing the tension applied to the FBG depending upon the temperature change, it is possible to adjust the grating period of the grating portion. As a result, it is possible to cancel the temperature dependency of the reflection center wavelength.

However, the above-mentioned temperature compensation device is disadvantageous in that the structure is complicated and the handling is difficult.

As a method for solving the above-mentioned problems, WO97/28480 discloses a method of controlling the tension applied to an FBG 15 by fixing the

FBG 15 to a glass ceramics substrate 14 obtained by heat treating and crystallizing a mother glass material preliminarily shaped into a plate and having a negative coefficient of thermal expansion, as illustrated in Fig. 2. In Fig. 2, the reference numeral 16 represents a grating portion, 17, an adhered and fixed portion, and 18, a weight.

Since the temperature compensation can be carried out by the single member, the method disclosed in WO97/28480 is simple in mechanism and easy to handle. However, the glass ceramics used therein is highly devitrifiable so that a resultant shape is restricted to a simple shape such as a plate. In other words, the member having a complicated shape can not be produced.

In addition, Japanese Unexamined Patent Publication JP 10-96827 A discloses a temperature compensation member made of a Zr-tungstate system material or a Hf-tungstate system material and having a negative coefficient of thermal expansion. However, since these materials are very expensive, it is difficult to put the disclosed one into practical use as an industrial product. Furthermore, in this temperature compensation member, the coefficient of thermal expansion is too large in a negative direction. This makes it difficult to successfully cancel the temperature dependency of the reflection center wavelength of the FBG. The coefficient of thermal expansion of the above-mentioned temperature compensation member can be adjusted in a positive direction by addition of a material, such as Al_2O_3 , having a positive coefficient of thermal expansion. However, addition of the material such as Al_2O_3 decreases the strength as a result of a large difference in expansion among the materials used. It is therefore difficult to put the disclosed one into practical use as an industrial product.

It is therefore an object of this invention to provide a temperature compensation member which has a negative coefficient of thermal expansion,

which can be shaped into even a complicated shape, and which can be manufactured at a low cost.

It is another object of this invention to provide an optical communication device using the above-mentioned temperature compensation member.

Disclosure of the Invention

In order to accomplish the above-mentioned object, the present inventors have made various experiments. As a result, it has been found out that, by sintering a large number of powder particles into a sintered body and including into the sintered body crystals exhibiting anisotropy in coefficient of thermal expansion, a temperature compensation member which has a negative coefficient of thermal expansion and which can be shaped into even a complicated shape can be manufactured at a low cost. Thus, this invention is proposed.

According to this invention, there is provided a temperature compensation member which comprises a sintered body obtained by firing at least one kind selected from a group including crystal powder, crystallizable glass powder, and partially-crystallized glass powder, which contains crystals exhibiting anisotropy in coefficient of thermal expansion, and which has a negative coefficient of thermal expansion.

According to this invention, there is provided a temperature compensation member which comprises a sintered body obtained by mixing at least one kind of powder selected from a group including crystal powder, crystallizable glass powder, and partially-crystallized glass powder and at least one additive selected from a group including amorphous glass powder, glass powder prepared by a sol-gel method, sol, and gel to obtain a mixture and firing the mixture, which contains crystals exhibiting anisotropy in coefficient of thermal expansion, and which has a negative coefficient of thermal expansion.

According to this invention, there is provided an optical communication device produced by the use of a temperature compensation member which comprises a sintered body obtained by firing at least one kind selected from a group including crystal powder, crystallizable glass powder, and partially-crystallized glass powder, which contains crystals exhibiting anisotropy in coefficient of thermal expansion, and which has a negative coefficient of thermal expansion.

According to this invention, there is provided an optical communication device produced by the use of a temperature compensation member which comprises a sintered body obtained by mixing at least one kind of powder selected from a group including crystal powder, crystallizable glass powder, and partially-crystallized glass powder and at least one additive selected from a group including amorphous glass powder, glass powder prepared by a sol-gel method, sol, and gel to obtain a mixture and firing the mixture, which contains crystals exhibiting anisotropy in coefficient of thermal expansion, and which has a negative coefficient of thermal expansion.

Brief Description of the Drawing

Fig. 1 is a front view showing an existing apparatus for preventing variation in reflection wavelength of an FBG in response to change in temperature.

Fig. 2 is a perspective view showing a glass ceramics substrate having a negative coefficient of thermal expansion with an FBG fixed to its surface.

Fig. 3 is a perspective view showing a ceramics sintered body as a temperature compensation member of this invention.

Best Mode of Embodying the Invention

A temperature compensation member according to this invention is prepared by accumulating or integrating and then sintering a large number of one kind or two or more kinds of powder selected from a group including crystal powder, crystallizable glass powder, and partially-crystallized glass powder. Therefore, even a complicated shape can be easily formed at a low cost by a forming technique such as pressing, casting, and extrusion.

In a method of obtaining a glass ceramic having a negative coefficient of thermal expansion by melting a glass material, forming a desired shape, and carrying out heat treatment to cause crystallization as in WO97/28480 mentioned above, a glass melt is highly devitrifiable and can not be formed into a complicated shape as will presently be described.

In order that a resultant glass ceramic has a negative coefficient of thermal expansion sufficient for temperature compensation, the degree of crystallinity must approximate to 100% and the composition of a precipitated crystal must approximate to that of a pure crystal. Therefore, it is inevitable that the composition of a mother glass is highly analogous to the crystal composition. The melt of such a mother glass is extremely highly devitrifiable. In every stage of a series of forming processes including injection from a nozzle, casting, roll-out, and cooling, coarse crystals are often deposited to cause a large difference in expansion in the glass. This results in easy occurrence of surface cracks during forming or machining. Therefore, it is impossible not only to produce a product complicated in shape but also to perform production at a yield of an industrial level.

On the other hand, in case where the crystal powder is used, it is unnecessary to melt the glass but production is possible by simply sintering the crystal powder produced by an existing technique. If the crystallizable glass powder or the partially-crystallized glass powder is used, the glass or the glass

ceramic is at first pulverized into powder and then sintered into a desired shape. Therefore, without taking devitrification of the molten glass into consideration, products having a complicated shape can be mass produced. Thus, use of the crystallizable glass powder or the partially-crystallized glass powder is free from the problem of decreasing the productivity because, even if the coarse crystals are deposited in the forming process, such coarse crystals are pulverized into fine particles in a pulverizing process to be homogenized.

Since the temperature compensation member of this invention contains crystals exhibiting anisotropy in coefficient of thermal expansion, a large number of microcracks are produced in a crystal grain boundary during cooling of crystal particles grown in a sintering process. Therefore, a negative coefficient of thermal expansion, specifically, -10 to $-120 \times 10^{-7}/^{\circ}\text{C}$ (preferably, -30 to $-90 \times 10^{-7}/^{\circ}\text{C}$) is obtained as a whole in a temperature range between -40 and 100°C . If the temperature compensation member is used for an FBG, the tension corresponding to the change in temperature is applied to the FBG to vary the grating period so that a component resulting from the variation in refractive index can be cancelled.

In this invention, respective crystal particles having anisotropic coefficients of thermal expansion expand or contract in various directions according to the coefficients of thermal expansion in their crystal axis directions. As a result, the crystal particles are rearranged to increase the filling density and to increase contact areas between the particles. This promotes the tendency such that the crystal particles are fused to one another during heat treatment to minimize the surface energy. As a result, a ceramics member having high strength, specifically, bending strength of 10 MPa or more is obtained. In this invention, the powder preferably has a particle size of $50\text{ }\mu\text{m}$ or less so as to increase the contact area between the powder particles.

It is noted here that the crystal exhibiting anisotropy in coefficient of thermal expansion is a crystal having a negative coefficient of thermal expansion in at least one crystal axis direction and a positive coefficient in other axis directions. As the crystal powder in this invention, use may be made of powder of silicate represented by β -eucryptite, titanate such as PbTiO_3 , phosphate such as $\text{NbZr}(\text{PO}_4)_3$, and oxide of La, Nb, V, or Ta. Among others, β -eucryptite crystal powder is suitable because the anisotropy in coefficient of thermal expansion is large. Furthermore, β -eucryptite crystal powder prepared by a so-called solid-phase method of mixing and firing a raw material powder is advantageous in that production at a low cost is possible because synthesis is carried out at a low temperature and pulverization is easy as compared with that prepared by a melting method in which the material is at first melted.

In this invention, it is preferable to mix the crystal powder and the crystallizable glass powder and/or the partially-crystallized glass powder prior to sintering. This is because the bending strength of the sintered body can be further improved. As regards the mixing ratio, 30-99 vol% of crystal powder and 1-70 vol% of crystallizable glass powder and/or partially-crystallized glass powder are appropriate.

In this invention, one kind or two or more kinds of powder selected from a group including crystal powder, crystallizable glass powder, and partially-crystallized glass powder and one kind or two or more kinds of additive selected from amorphous glass powder, glass powder prepared by a sol-gel method, sol, and gel may be mixed and then sintered. In this case, the firing temperature is lowered so as to achieve the improvement in workability and the reduction in cost. As regards the mixing ratio, 50-99.9 vol% of one kind or two or more kinds of crystal powder, crystallizable glass powder, and partially-crystallized glass powder and 0.1-50 vol% of one kind or two or more kinds of additive are

appropriate.

It is noted here that the crystallizable glass powder is glass powder having a property such that crystals are precipitated inside as a result of heat treatment. The partially-crystallized glass powder is glass ceramic powder such that the crystals have been precipitated in the glass. In this invention, another kind of crystal powder (for example, Al_2O_3 powder) different from the above-mentioned crystal powder may be mixed. In this event, the effects of further facilitating adjustment of the coefficient of thermal expansion, the strength, or the chemical properties is obtained.

In the temperature compensation member of this invention, a sintered body of a complicated shape can readily be prepared by a forming technique such as pressing, casting, and extrusion as described above. For example, a groove or a through hole for disposing an optical device can be easily formed at a predetermined position of the sintered body. In manufacture of an optical communication device, this provides greater advantages as follows.

For example, an optical fiber of an FBG is adhered and fixed to the temperature compensation member by the use of an adhesive. If the temperature compensation member has a groove or a through hole formed at a predetermined position thereof to locate an optical device, assembling is easily automated when the optical fiber is adhered. Therefore, the production cost is lowered. The groove or the through hole is not restricted to a single position but may be formed at a plurality of positions.

Generally, when a fiber-shaped optical device such as the FBG is fixed to the temperature compensation member, the optical device must preliminarily be applied with tension so that the optical device is not bent in case where the temperature compensation member is contracted to a length shorter than that when it is fixed. If the groove or the through hole has a diameter close to that of the optical device, the amount of the adhesive to be used can be reduced and

the fixation can be achieved by a thin adhesive layer. Such a thin adhesive layer decreases a stress due to the difference in thermal expansion between the adhesive and each of the optical device and the temperature compensation member. This allows adhesion and fixation throughout the entire length of the groove or the through hole. In this event, even if the temperature compensation member contracts to the length shorter than that when it is fixed, the optical device is prevented from being bent or loosened. Therefore, the optical device with a temperature compensating function can be produced in a simpler process without requiring the tension to be preliminarily applied. In particular, in case where the temperature compensation member is provided with a precise through hole to receive the optical device to be inserted therethrough, the temperature compensation member also has a function as a component for positioning the optical device. Thus, the temperature compensation member itself serves as a connector component when the device with the temperature compensating function is connected to an optical fiber or another device.

As the adhesive for use in adhesion of the optical device to the temperature compensation member of this invention, low-melting-point glass frit or epoxy resin is suitable. Particularly, an adhesive comprising an alkali silicate aqueous solution (specifically, sodium silicate aqueous solution, potassium silicate aqueous solution) and inorganic powder (specifically, $\text{Li}_2\text{O}-\text{Al}_2\text{O}_3-\text{SiO}_2$ system glass ceramic powder with β -spodumene, β -spodumene solid solution, β -eucryptite, or β -quartz solid solution precipitated therein) is advantageous because long-term stability is excellent and adhesion is possible at a low temperature.

Now, this invention will be described in detail in conjunction with various examples and comparative examples.

(Example 1)

At first, β -eucryptite crystals were pulverized to obtain crystal powder having an average particle size of $10\ \mu\text{m}$ or less. Thereafter, the crystal powder was put into a mold and press-formed at a temperature of 20 MPa to produce a molded body (powder compact) 19 having a rectangular prism shape of 4mm wide, 3mm thick, and 40mm long with a groove 19a of 1mm wide and 1mm deep formed on an upper surface thereof at the center in a longitudinal direction, as illustrated in Fig. 3.

Then, the molded body 19 was fired in air at 1300°C for 2 hours to be sintered, and thereafter cooled down to a room temperature. Thus, a ceramics sintered body comprising β -eucryptite with a number of microcracks formed at a crystal grain boundary was obtained.

(Example 2)

$\text{Pb}_{0.9}\text{Ca}_{0.1}(\text{Fe}_{0.5}\text{Nb}_{0.5})_{0.5}\text{Ti}_{0.5}\text{O}_3$ crystals were pulverized to obtain crystal powder having an average particle size of $10\ \mu\text{m}$ or less. The crystal powder was press-formed in the manner similar to Example 1 to produce a molded body. The molded body was fired in air at 1320°C for 1 hour to be sintered. Thus, a ceramics sintered body comprising $\text{Pb}_{0.9}\text{Ca}_{0.1}(\text{Fe}_{0.5}\text{Nb}_{0.5})_{0.5}\text{Ti}_{0.5}\text{O}_3$ with a number of microcracks formed at a crystal grain boundary was obtained.

(Example 3)

At first, β -eucryptite crystals were pulverized to obtain crystal powder having an average particle size of $10\ \mu\text{m}$ or less. Then, the crystal powder was mixed with 35%, by volume, of glass powder having the same average particle size, comprising SiO_2 , Al_2O_3 , and MgO as main components, and capable of precipitating cordierite when heated. Thereafter, press forming was performed in the manner similar to Example 1 to produce a molded body. The molded body was fired in air at 1300°C for 10 hours to be sintered. Thus, a sintered body containing β -eucryptite solid solution with a number of microcracks

formed at a crystal grain boundary was obtained.

(Example 4)]

At first, $\text{NbZr}(\text{PO}_4)_3$ crystals were pulverized to obtain crystal powder having an average particle size of $10\ \mu\text{m}$ or less. Then, the crystal powder was mixed with 10% of Al_2O_3 powder having the same average particle size to obtain mixed powder. The mixed powder with water added thereto was kneaded into a slurry, poured into a gypsum mold having a predetermined shape, dried, and removed from the mold to produce a cast body. The cast body was fired in air at 1350°C for 5 hours to be sintered. Thus, a sintered body similar in shape to Example 1 and containing $\text{NbZr}(\text{PO}_4)_3$ with a number of microcracks formed at a crystal grain boundary was obtained.

(Example 5)

At first, β -eucryptite crystals were pulverized to obtain crystal powder having an average particle size of $10\ \mu\text{m}$ or less. Then, the crystal powder was mixed with 35%, by volume, of glass powder having the same average particle size, comprising SiO_2 , Al_2O_3 , and Li_2O as main components, and capable of precipitating β -eucryptite solid solution or β -spodumene solid solution when heated, to obtain mixed powder. The mixed powder was cast in the manner similar to Example 4 to produce a cast body. The cast body was fired in air at 1300°C for 2 hours to be sintered. Thus, a sintered body containing β -eucryptite solid solution with a number of microcracks formed at a crystal grain boundary was obtained.

(Example 6)

At first, β -eucryptite crystals were pulverized to obtain crystal powder having an average particle size of $10\ \mu\text{m}$ or less. Then, the crystal powder was mixed with 30%, by volume, of $\text{NbZr}(\text{PO}_4)_3$ crystals having the same average particle size to obtain mixed powder. The mixed powder was press-formed in the manner similar to Example 1 to produce a molded body. The molded body

was fired in air at 1300°C for 5 hours to be sintered. Thus, a sintered body containing β -eucryptite crystals and $\text{NbZr}(\text{PO}_4)_3$ crystals with a number of microcracks formed at a crystal grain boundary was obtained.

(Example 7)

At first, a glass material was prepared to have a composition of 46% SiO_2 , 41% Al_2O_3 , 9% Li_2O , 1% TiO_2 , and 3% ZrO_2 by weight percent. The glass material was melted in a platinum crucible at 1550°C for 6 hours, granulated in water, pulverized by a ball mill, and classified to obtain crystallizable glass powder having an average particle size of 10 μm .

Then, the crystallizable glass powder was press-formed in the manner similar to Example 1 to obtain a molded body. The molded body was heated at 1350°C for 10 hours and thereafter cooled at a temperature falling rate of 200°C/hour to obtain a ceramics sintered body..

(Example 8)

At first, a water-granulated glass having a composition similar to that in Example 7 was heated at 900°C for 1 hour and then cooled at a temperature falling rate of 200°C/hour to obtain a partially-crystallized glass containing β -quartz solid solution crystals precipitated inside and having the degree of crystallinity of about 80%.

Then, the partially-crystallized glass was pulverized by a ball mill and classified to obtain partially-crystallized glass powder having an average particle size of 10 μm . The glass powder was press-formed in the manner similar to Example 1 to produce a molded body. The molded body was heated at 1350°C for 10 hours and thereafter cooled at a temperature falling rate of 200°C/hour to obtain a ceramics sintered body.

(Example 9)

β -eucryptite crystals were pulverized to obtain crystal powder having an average particle size of 10 μm . On the other hand, preparation was made of

amorphous glass powder (average particle size of $10\ \mu\text{m}$) having a composition of 63% SiO_2 , 6% Na_2O , 6% Al_2O_3 , 20% B_2O_3 , 2% K_2O , and 3% BaO by weight percent. Thereafter, 85 vol% of the crystal powder and 15 vol% of the amorphous glass powder were mixed, put into a mold, and press-formed at a pressure of 20 MPa to obtain a molded body similar to that in Example 1.

Then, the molded body was fired in air at 1000°C for 1 hour to be sintered. Thus, a ceramics sintered body containing β -eucryptite crystals with a number of microcracks formed in a crystal phase was obtained.

(Example 10)

$\text{Pb}_{0.9}\text{Ca}_{0.1}(\text{Fe}_{0.5}\text{Nb}_{0.5})_{0.5}\text{Ti}_{0.5}\text{O}_3$ crystals were pulverized to obtain crystal powder having an average particle size of $10\ \mu\text{m}$. On the other hand, preparation was made of crystallizable glass powder (average particle size of $10\ \mu\text{m}$) having a composition of 65% SiO_2 , 22% Al_2O_3 , 5% Li_2O , 2% K_2O , 2% P_2O_5 , 1% MgO and 3% ZnO by weight percent and capable of precipitating β -quartz solid solution crystals inside when heated.

Thereafter, 85 vol% of the crystal powder and 15 vol% of the crystallizable glass powder were mixed, put into a mold, and press-formed at a pressure of 20 MPa to obtain a molded body similar to that in Example 1.

Then, the molded body was fired in air at 1200°C for 3 hours to be sintered. Thus, a ceramics sintered body containing

$\text{Pb}_{0.9}\text{Ca}_{0.1}(\text{Fe}_{0.5}\text{Nb}_{0.5})_{0.5}\text{Ti}_{0.5}\text{O}_3$ crystals and β -eucryptite crystals with a number of microcracks formed in a crystal phase was obtained.

(Example 11)

Preparation was made of β -quartz solid solution powder having an average particle size of $10\ \mu\text{m}$. On the other hand, preparation was made of crystallizable glass powder (average particle size of $10\ \mu\text{m}$) having a composition of 67% SiO_2 , 23% Al_2O_3 , 5% Li_2O , 1.4% P_2O_5 , 2.3% ZrO_2 and 1.3%

Then, the molded body was fired in air at 1200°C for 5 hours to be sintered. Thus, a ceramics sintered body having a β -quartz solid solution crystal phase with a number of microcracks was obtained.

80 vol% of β -eucryptite crystal powder similar to that in Example 1 and 20 vol% of SiO₂ glass powder (average particle size of 5 μ m) prepared by a sol-gel method were mixed to obtain mixed powder. The mixed powder with water added thereto was kneaded into a clay-like material and then subjected to extrusion to produce a tubular molded body having an outer diameter of 3mm and an inner diameter of 0.3mm.

Then, the molded body was fired in air at 1200°C for 12 hours to be sintered. Thus, a ceramics sintered body containing a number of β -eucryptite crystals with a number of microcracks formed in a crystal was obtained.

60 wt% of β -eucryptite crystal powder similar to that in Example 1 and 40 wt% of $\text{Al}(\text{OC}_4\text{H}_9)_3$ solution having a concentration of 10% were mixed to obtain a mixed material. The mixed material was dried at a temperature of 120°C, put into a mold, and press-formed at a temperature of 20 MPa to obtain a molded body similar to that in Example 1.

Then, the molded body was fired in air at 900°C for 5 hours to be sintered. Thus, a ceramics sintered body containing β -eucryptite crystals and alumina crystals with a number of microcracks formed in a crystal phase was obtained.

(Example 14)

80 vol% of $\text{NbZr}(\text{PO}_4)_3$ crystals having an average particle size of $15 \mu\text{m}$ and 20 vol% of amorphous glass powder (average particle size of $10 \mu\text{m}$) comprising 65% SiO_2 , 6% Al_2O_3 , 1% Li_2O , 20% B_2O_3 , 3% BaO , 0.5% F , 2.5% Na_2O , and 2% K_2O by weight percent were mixed to obtain mixed powder. The mixed powder was put into a mold and press-formed at a pressure of 20 MPa to obtain a molded body similar to that in Example 1.

Then, the molded body was fired in air at 1100°C for 2 hours to be sintered. Thus, a ceramics sintered body containing $\text{NbZr}(\text{PO}_4)_3$ crystals with a number of microcracks formed in a crystal was obtained.

(Example 15)

50 vol% of SnO_2 crystal powder having an average particle size of $5 \mu\text{m}$ and 50 vol% of partially-crystallized glass powder with 80 vol% of β -eucryptite crystals precipitated therein were mixed to obtain mixed powder. The mixed powder was put into a mold and press-formed at a pressure of 20 MPa to obtain a molded body similar to that in Example 1.

Then, the molded body was fired in air at 1300°C for 10 hours to be sintered. Thus, a ceramics sintered body containing SnO_2 crystals and β -eucryptite crystals with a number of microcracks formed in a crystal was obtained.

(Example 16)

55 vol% of β -eucryptite crystal powder similar to that in Example 1 and crystallizable glass powder (average particle size of $10 \mu\text{m}$) having a composition of 65% SiO_2 , 22% Al_2O_3 , 5% Li_2O , 2% K_2O , 2% P_2O_5 , 1% MgO and 3% ZnO by weight percent and capable of precipitating β -quartz solid solution crystals inside when heated were mixed to obtain mixed powder. The mixed powder was put into a mold and press-formed at a pressure of 20 MPa to obtain a molded body similar to that in Example 1.

Then, the molded body was fired in air at 1250°C for 5 hours to be sintered. Thus, a ceramics sintered body containing β -eucryptite crystals with a number of microcracks formed in a crystal phase was obtained.

(Comparative Example 1)

A glass melt in which a mol ratio of $\text{Li}_2\text{O} : \text{Al}_2\text{O}_3 : \text{SiO}_2$ is 1 : 1 : 2 was poured into a mold, cooled, formed into a shape similar to that in Example 1, and fired at 1300°C for 15 hours to obtain a glass ceramic comprising β -eucryptite crystals with a number of microcracks contained in a crystal phase.

(Comparative Example 2)

60 vol% of SnO_2 powder having an average particle size of 10 μm and 40 vol% of glass powder having an average particle size of 10 μm , comprising SiO_2 , Al_2O_3 , and Li_2O as main components, and capable of precipitating β -quartz solid solution or β -spodumene solid solution when heated were mixed to obtain mixed powder. The mixed powder was put into a mold and press-formed at a pressure of 20 MPa into a shape similar to that in Example 1 to produce a molded body. The molded body was fired in air at 1400°C for 15 hours to be sintered. Thus, a ceramics sintered body was obtained. The sintered body contained SnO_2 crystals but no microcracks were formed in a crystal phase.

(Comparative Example 3)

SnO_2 powder having an average particle size of 5 μm was press-formed into a shape similar to that in Example 1 to obtain a molded body. The molded body was fired in air at 1400°C for 15 hours to be sintered. Thus, a ceramics sintered body was obtained. The sintered body contained SnO_2 crystals but no microcracks were formed in a crystal.

The ceramics sintered bodies in Examples and Comparative Examples obtained as mentioned above were measured for the coefficient of thermal expansion and the bending strength. The result is shown in Table 1.

Table 1

	Coefficient of Thermal Expansion ($\times 10^{-7}/^{\circ}\text{C}$)	Bending Strength (MPa)	Formability
Example 1	-80	15	good
Example 2	-45	20	good
Example 3	-66	30	good
Example 4	-51	25	good
Example 5	-78	30	good
Example 6	-60	25	good
Example 7	-69	28	good
Example 8	-69	28	good
Example 9	-72	45	good
Example 10	-55	30	good
Example 11	-45	20	good
Example 12	-85	35	good
Example 13	-80	20	good
Example 14	-40	40	good
Example 15	-30	20	good
Example 16	-80	20	good
Comparative Example 1	-80	20	no good
Comparative Example 2	+30	25	good
Comparative Example 3	+40	20	good

As is clear from Table 1, the ceramics sintered body in each Example has a negative coefficient of thermal expansion between -30 to $-85 \times 10^{-7}/^{\circ}\text{C}$ and a high bending strength of 15 MPa or more. Furthermore, the ceramics sintered body is suitable as the temperature compensation member used in the FBG because a groove having a predetermined shape is formed.

On the other hand, the glass ceramic in Comparative Example 1 exhibits remarkable devitrification upon forming to deposit coarse crystals and to form a large number of cracks on its surface. The ceramics sintered bodies in each of Comparative Examples 2 and 3 has a positive coefficient of thermal expansion and can not be used as the temperature compensation member.

It is noted here that the coefficients of thermal expansion in Table 1 were measured by a dilatometer in a temperature range between -40 and 100°C . The bending strength was measured by a three-point bending test according to

JIS R1601 after each ceramics sintered body is shaped into a plate of 3mm x 4mm x 35mm. As regards the formability, "good" represents the case where the molded body shown in Fig. 1 was accurately prepared while "no good" represents the case where the molded body could not accurately be prepared and had cracks formed on its surface. Identification of the crystal phase was examined by X-ray diffraction. By the use of a scanning electron microscope, presence or absence of microcracks was observed.

The temperature compensation member in each of the above-mentioned Examples 1 to 16 has a negative coefficient of thermal expansion and, even if the shape is complicated, can easily be formed at a low cost.

Industrial Applicability

The temperature compensation member of this invention is suitable as a temperature compensation member of an optical communication device such as an FBG, a coupler, and a waveguide.

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Claims

1. A temperature compensation member which comprises a sintered body obtained by firing at least one kind selected from a group including crystal powder, crystallizable glass powder, and partially-crystallized glass powder, which contains crystals exhibiting anisotropy in coefficient of thermal expansion, and which has a negative coefficient of thermal expansion.

2. A temperature compensation member according to claim 1, wherein said crystal powder is at least one kind of powder selected from a group including silicate, phosphate, titanate, and oxides of La, Nd, V, and Ta.

3. A temperature compensation member according to claim 1, wherein said crystal powder is β -eucryptite crystal powder prepared by a solid-phase method.

4. A temperature compensation member according to claim 1, wherein said powder has an average particle size of 50 μm or less.

5. A temperature compensation member according to claim 1, wherein the coefficient of thermal expansion falls within a range of -10 to $-120 \times 10^{-7}/^{\circ}\text{C}$ in a temperature range of -40 to 100°C .

6. A temperature compensation member according to claim 1, which comprises a sintered body obtained by mixing and firing said crystal powder and at least one of said crystallizable glass powder and said partially-crystallized glass powder, which contains crystals exhibiting anisotropy in coefficient of thermal expansion, and which has a negative coefficient of thermal expansion.

7. A temperature compensation member which comprises a sintered body obtained by mixing at least one kind of powder selected from a group including crystal powder, crystallizable glass powder, and partially-crystallized glass powder and at least one additive selected from a group including amorphous glass powder, glass powder prepared by a sol-gel method, sol, and

gel to obtain a mixture and firing the mixture, which contains crystals exhibiting anisotropy in coefficient of thermal expansion, and which has a negative coefficient of thermal expansion.

8. An optical communication device produced by the use of a temperature compensation member which comprises a sintered body obtained by firing at least one kind selected from a group including crystal powder, crystallizable glass powder, and partially-crystallized glass powder, which contains crystals exhibiting anisotropy in coefficient of thermal expansion, and which has a negative coefficient of thermal expansion.

9. An optical communication device according to claim 8, said device being produced by the use of a temperature compensation member which comprises a sintered body obtained by mixing and firing said crystal powder and at least one of said crystallizable glass powder and said partially-crystallized glass powder, which contains crystals exhibiting anisotropy in coefficient of thermal expansion, and which has a negative coefficient of thermal expansion.

10. An optical communication device produced by the use of a temperature compensation member which comprises a sintered body obtained by mixing at least one kind of powder selected from a group including crystal powder, crystallizable glass powder, and partially-crystallized glass powder and at least one additive selected from a group including amorphous glass powder, glass powder prepared by a sol-gel method, sol, and gel to obtain a mixture and firing the mixture, which contains crystals exhibiting anisotropy in coefficient of thermal expansion, and which has a negative coefficient of thermal expansion.

Abstract

A member comprises a sintered body obtained by firing one kind or two or more kinds selected from a group including crystal powder, crystallizable glass powder, and partially-crystallized glass powder and has a negative coefficient of thermal expansion. The member contains crystals exhibiting anisotropy in coefficient of thermal expansion.

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FIG. 1

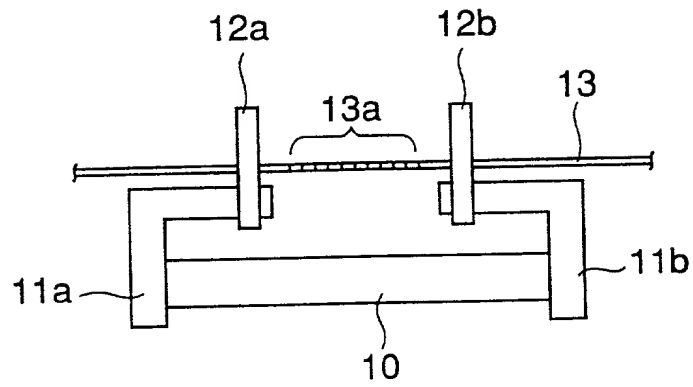


FIG. 2

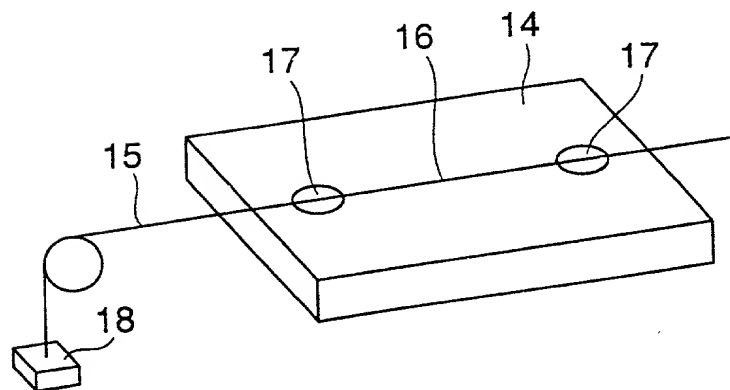
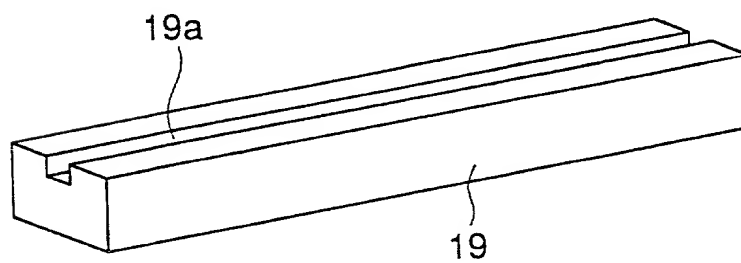


FIG. 3



As a below named inventor, I hereby declare that:

G-1527
米田 宣彦

My residence, post office address and citizenship are as stated below next to my name,

I believe I am the original, first and sole inventor (if only one name is listed below) or an original, first and joint inventor (if plural names are listed below) of the subject matter which is claimed and for which a patent is sought on the invention entitled:

TEMPERATURE COMPENSATION MEMBER AND OPTICAL
COMMUNICATION DEVICE USING THE SAME

the specification of which (check only one item below):

- ☐ is attached hereto.
- ☐ was filed as United States application
Serial No. _____
on _____
and was amended
on _____ (if applicable).
- ☒ was filed as PCT international application
Number PCT/JP00/00714
on February 9, 2000
and was amended under PCT Article 19
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I hereby state that I have reviewed and understand the contents of the above-identified specification, including the claims, as amended by any amendment referred to above.

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I hereby claim foreign priority benefits under Title 35, United States Code, §119 of any foreign application(s) for patent or inventor's certificate or of any PCT international application(s) designating at least one country other than the United States of America listed below and have also identified below any foreign application(s) for patent or inventor's certificate or any PCT international application(s) designating at least one country other than the United States of America filed by me on the same subject matter having a filing date before that of the application(s) of which priority is claimed:

PRIOR FOREIGN/PCT APPLICATION(S) AND ANY PRIORITY CLAIMS UNDER 35 U.S.C. 119:

COUNTRY (if PCT, indicate "PCT")	APPLICATION NUMBER	DATE OF FILING (day, month, year)	PRIORITY CLAIMED UNDER 35 USC 119
Japan	47022/1999	24 February 1999	<input checked="" type="checkbox"/> YES <input type="checkbox"/> NO
Japan	52780/1999	1 March 1999	<input checked="" type="checkbox"/> YES <input type="checkbox"/> NO
Japan	52793/1999	1 March 1999	<input checked="" type="checkbox"/> YES <input type="checkbox"/> NO
Japan	332577/1999	24 November 1999	<input checked="" type="checkbox"/> YES <input type="checkbox"/> NO
			<input type="checkbox"/> YES <input type="checkbox"/> NO

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PCT APPLICATION NO.	PCT FILING DATE	U.S. SERIAL NUMBERS ASSIGNED (if any)			

POWER OF ATTORNEY: As a named inventor, I hereby appoint the following attorney(s) and/or agent(s) to prosecute this application and transact all business in the Patent and Trademark Office connected therewith. (List name and registration numbers):

ALLISON C. COLLARD, Registration No. 22,532; KURT KELMAN, Registration No. 18,628
EDWARD R. FREEDMAN, Registration No. 26,048; FREDERICK J. DORCHAK, Registration No. 29,298
ELIZABETH COLLARD RICHTER, Registration No. 35,103; WILLIAM C. COLLARD, Registration No. 38,411
REINE H. GLANZ, Registration No. 46,728

Send Correspondence to: COLLARD & ROE, P.C.
1077 Northern Boulevard
Roslyn, New York 11576

Customer No. **25889**

Direct Telephone Calls to:
(name and telephone number)
(516) 365-9802

1	FULL NAME OF INVENTOR	FAMILY NAME MATANO	FIRST GIVEN NAME TAKAHIRO	SECOND GIVEN NAME
	RESIDENCE & CITIZENSHIP	CITY Echi-gun, Shiga	STATE OR FOREIGN COUNTRY Japan JAX	COUNTRY OF CITIZENSHIP Japan
	POST OFFICE ADDRESS	POST OFFICE ADDRESS c/o NIPPON ELECTRIC GLASS CO., LTD., 7-1, Seiran 2-chome		CITY Otsu-shi, Shiga
2	FULL NAME OF INVENTOR	FAMILY NAME SAKAMOTO	FIRST GIVEN NAME AKIHIKO	SECOND GIVEN NAME
	RESIDENCE & CITIZENSHIP	CITY Koka-gun, Shiga	STATE OR FOREIGN COUNTRY Japan JAX	COUNTRY OF CITIZENSHIP Japan
	POST OFFICE ADDRESS	POST OFFICE ADDRESS c/o NIPPON ELECTRIC GLASS CO., LTD., 7-1, Seiran 2-chome		CITY Otsu-shi, Shiga
3	FULL NAME OF INVENTOR	FAMILY NAME	FIRST GIVEN NAME	SECOND GIVEN NAME
	RESIDENCE & CITIZENSHIP	CITY	STATE OR FOREIGN COUNTRY	COUNTRY OF CITIZENSHIP
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SIGNATURE OF INVENTOR 201 <i>Takahiro Matano</i>	SIGNATURE OF INVENTOR 202 <i>Akihiko Sakamoto</i>	SIGNATURE OF INVENTOR 203
DATE August 7, 2001	DATE August 7, 2001	DATE